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CONTRIBUTIONS FROM THE CHEMICAL LABORATORY  
OF HARVARD COLLEGE.

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*A REVISION OF THE ATOMIC WEIGHT OF NICKEL.*

FIRST PAPER.—THE ANALYSIS OF NICKELOUS BROMIDE.

BY THEODORE WILLIAM RICHARDS AND ALLERTON SEWARD CUSHMAN.



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Presented June 9, 1897.

INTRODUCTION.

Of the atomic weights to-day those of nickel and cobalt are among the most interesting, not only because the two values are so close together, but also because the purely elementary character of these metals has been recently doubted.\* A careful study of the available literature upon the subject leaves the impartial critic in grave doubt as to the true values to be accepted, hence an experimental revision seems to be imperative; and the present work is a part of a comprehensive attempt to conduct such a revision. The very useful and complete index of the work which has been done upon this subject, recently published by Professor Clarke,† makes a detailed statement of the various researches unnecessary here; but a chronological list is appended.

THE ATOMIC WEIGHT OF NICKEL.

O = 16.

1826. By Analysis of Chloride.	
Rothoff . . . . .	59.1
1852. Reduction of the Oxide by Hydrogen.	
Erdman and Marchand . . . . .	58.2-58.6
1856. Conversion of Nickel to Sulphate.	
Deville . . . . .	58.85
1857. Analysis of the Oxalate.	
Schneider . . . . .	58.07
1858. Analysis of the Sulphate and Chloride.	
Marignac . . . . .	58.4-59.29

\* Zeit. Anorg. Chem., II. 235; Berichte d. d. ch. Gesell., 1889, pp. 11, 2026.

† Recalculation of the Atomic Weights, Smithsonian. Misc. Coll., Constants of Nature, Part V., 1897, p. 291.

1860. Synthesis of the Chloride.	
Dumas . . . . .	59.02
1863. Reduction of the Oxides in Hydrogen.	
Russell . . . . .	58.74
1866. Analysis of Nickel Potassic Sulphate.	
Sommaruga . . . . .	58.03
1867. By relation to Gold, out of Sodic Auro-chloride.	
Winkler . . . . .	59.45
1867. Measurement of Hydrogen evolved by acting on Nickel with HCl.	
Russell . . . . .	58.77
1871. Analysis of Strychnine and Brucine Nickelo-cyanides.	
Lee . . . . .	58.01
1883. Analysis of the Sulphate.	
Baubigny . . . . .	58.73
1886. Reduction of Oxide (NiO) by Hydrogen.	
Zimmermann . . . . .	58.71
1890. Reduction of Oxide by Hydrogen.	
Mond, Langer, and Quincke . . . . .	58.58
1892. Conversion of Sulphate to Oxide, and Reduction in Hydrogen.	
Schützenberger . . . . .	58.54
1892. Krüss and Schmidt . . . . .	57-64
1893-94. Analysis of Nickelous Chloride and by relation to Iodine.	
Winkler , . . . .	59.05-58.87
Value accepted by Ostwald,* 1890 . . .	Ni = 58.5
"        "    Clarke,† 1896 . . .	Ni = 58.687
"        "    Seubert,‡ 1896 . . .	Ni = 58.9

A glance at this list shows the lack of consistency in the results obtained. In many cases this is sufficiently accounted for by the inadequacy of the methods, and the known impurity of the materials used, without taking into consideration the unconfirmed admixture of unknown impurities. A critical investigation of the data already published would be an endless and perhaps unprofitable labor, and we prefer at this time rather to record the method and results of our own work than to enter into any discussion of claims heretofore advanced.

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\* Lehrbuch, Vol. I. p. 96.

† Recalculation of the Atomic Weights, *loc. cit.*

‡ Zeitschr. Anorg. Chem., XIII. 229.

While it is obvious that certainty is to be obtained only by securing unanimous results from a number of compounds and methods, it is equally obvious that a single method, well worked out, is far better than twenty incomplete ones; hence the present work was confined for the present to a single compound.

In our choice of material, we were guided by experience already gained in this Laboratory. The advantage of the bromides as typical compounds in atomic weight determinations has been discussed by one of us in preceding publications, and requires no further mention. The rather meagre current descriptions of the properties of anhydrous nickel bromide did not seem encouraging, but the progress of the research showed that it is possible to dry this salt, weigh it, and dissolve it in water in a perfectly normal condition. Since this was the case, nickelous bromide was naturally chosen as our starting point.

#### THE PREPARATION AND PROPERTIES OF NICKELOUS BROMIDE.

Finely divided nickel, when heated to a red heat in a stream of dry bromine vapor, readily forms the bromide, which sublimes at bright redness. The color of the sublimate varies from a pale straw-yellow to a dark bronze-brown, according to the state of aggregation. At a red heat in the presence of traces of air or moisture, the salt loses traces of bromine with the formation of bright green nickelous oxide, unless much hydrobromic acid is present. We have never obtained evidence that an oxybromide is formed under these conditions.

The sublimed bromide is almost insoluble in cold water, but solution soon becomes apparent to the eye in water at 50°. In water at 90° the salt dissolves less slowly, but a gram still requires an hour or two for its complete solution. When originally free from oxide, the sublimed bromide dissolves in water even at the boiling point to form a solution of perfect clearness. According to Berthelot,\* a solution of nickelous bromide left for some time in contact with air deposits some flakes of the oxide. We have never met with the slightest evidence of the truth of this statement in the case of a dilute solution. If, however, the nickelous bromide contained only a slight admixture of the oxide, this oxide might escape observation until it had settled out upon the bottom of the vessel. Dilute nitric acid does not materially hasten the solution of the bromide. The sublimed crystalline salt is hygroscopic in character, although not nearly to the extent which we had been led to expect. From several

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\* Anal. Chim. Phys., [2], XLIV. 389.

experiments carried out to test this point it appeared that freshly sublimed nickelous bromide exposed to the free air of the room absorbed about 0.1 milligram per gram in ten minutes.

Since the value of the selection of nickelous bromide as the starting point in the determination of the atomic weight of nickel depends on the possibility of dissolving the salt in hot water without the slightest loss of bromine, it seemed necessary to investigate this point. To this end, a mass of several grams of the sublimed compound was suspended in a flask arranged in such a way that the atmosphere of the flask during the solution could be swept through a bulb-tube containing a mixture of potassic iodide and starch. Any formation of nickelous oxide or oxybromide during the heating of the solution could have taken place only by disengagement of bromine, which would have been shown by the blueing of the indicator in the bulb. Not the slightest tinge of blue color appeared at any time during the experiment, in spite of the fact that the solution of nickel bromide was finally subjected to prolonged boiling. That the iodo-starch mixture was exceedingly sensitive to very minute traces of free bromine was assured by experiment in the first place. This experiment is detailed here, however, merely as corroborative testimony, and is not advanced as being final on the subject; the best evidence is yielded by the quantitative results of our series of analyses.

Being now sure that we could analyze the salt if we could obtain it in a pure state, we next proceeded to determine whether it could be prepared in a condition altogether suitable for weighing and analysis. The first sublimations were performed in an ordinary combustion tube in an atmosphere of carbon dioxide. These sublimations yielded in no case material that was above suspicion, and none of it was used in our analyses, the experiments being carried out merely as a study of the properties of the substance under examination. Since it seemed possible that the carbon dioxide used might suffer partial dissociation at the high temperature necessary for the sublimation, nitrogen was substituted for this gas. After many experiments with nitrogen, both alone and when mixed with bromine vapor, it was found best to carry on the sublimation of the nickelous bromide in a stream of nitrogen mixed with hydrobromic acid. The elaborate apparatus constructed by Mr. G. P. Baxter for supplying suitable mixtures of any or all of these gases and vapors for preparing and subliming the salt will be described in a later paper, upon the atomic weight of cobalt.

The temperature at which the salt sublimes lies not far from that at which the hardest glass begins to soften, hence we found it advantageous

to conduct the sublimation in porcelain. In order to collect the product a small porcelain tube, used as the receiver, was fitted, telescope fashion, inside of the larger tube containing the substance to be vaporized. In this way the pure crystals could be removed without contamination. Since nickelous bromide is decomposed either by oxygen or by water at a red heat, unless a very large excess of hydrobromic acid gas is present, both oxygen and water must be excluded with scrupulous care during its final sublimation. At first we were not able to accomplish this complete exclusion, so that most of the bromide used in the preliminary series of analyses contained traces of green crystalline oxide, which were carefully collected and weighed. In making the final calculation, the weight of this oxide was subtracted from the total, in order to obtain the weight of the bromide. Subsequently, with improved apparatus, the bromide

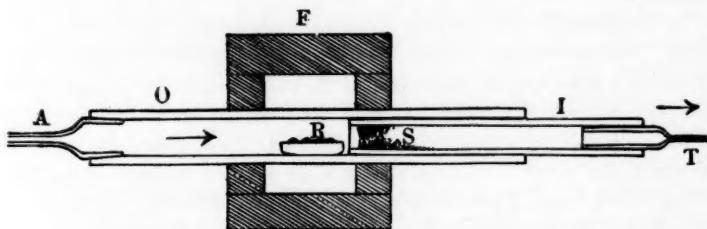


FIG. 1. SECTION OF APPARATUS FOR SUBLIMATION.

A = glass tube for admitting bromine vapor. O = outer porcelain tube. I = inner porcelain tube. T = glass outlet tube. F = perforated Fletcher furnace. R = boat containing nickel. S = Sublimed bromide.

was obtained in a state of satisfactory purity; it gave a perfectly clear solution in water, leaving nothing to be desired. With the improved arrangement the oxide itself was quickly converted at a dull red heat into the bromide, by means of a stream of hydrobromic acid gas. Hence it was possible to purify nickelous bromide contaminated with oxide by simply treating it for a few minutes in this fashion; in practice, the scheme worked very well.

Chemical literature contains no satisfactory determination of the specific gravity of the anhydrous salt; hence, in order to reduce our weighings to the vacuum standard, the following determination was made. 3.3196 grams of pure dry nickelous bromide displaced upon one occasion 0.6162 gram and upon another 0.6156 gram of rectified toluol at 28°. Since the specific gravity of the toluol at this temperature was 0.860,



referred to water at the same temperature, the specific gravity of the bromide must be 4.64. Nickelous bromide is insoluble in toluol.

#### PURIFICATION OF MATERIALS.

*Nickel.* — Since the possibility of preparing and analyzing pure nickelous bromide had now been proved, the next step was to purify all the materials concerned in its manufacture. First among these materials comes nickel, which must not only be freed from all known impurities, but must also be so treated as to detect and eliminate unknown ones. With this latter purpose in view, our material was obtained from two distinct sources; first, the "pure" nickel of commerce; and, secondly, really pure nickel (containing only a little iron) prepared by Dr. Mond through the carbonic oxide process and kindly presented by Dr. Wolcott Gibbs.

It is convenient to consider first our treatment of the commercial nickel. In proceeding with the further purification of this sample, our first step was to remove the metals of the copper and tin groups. The simple treatment with hydric sulphide has generally been considered sufficient to insure the separation of the metals of these groups, in spite of the fact that many of the sulphides when present in small quantities often assume a colloidal condition in which they cannot be separated by filtration. In our case this difficulty was avoided by regulating the acidity of the solution so that a certain amount of black nickelous sulphide was precipitated, which effectually "swept" this liquid, coagulating small quantities of foreign sulphides. After filtration the liquid was boiled to drive off the hydric sulphide, oxidized with a few drops of nitric acid, made alkaline with ammonia, and filtered. The precipitation of the sulphide was now continued by the passage of a little washed hydric sulphide. This first comparatively small amount of sulphide was filtered out and discarded. The remaining nickel was then as nearly as possible completely precipitated in the form of sulphide. After the precipitate had been allowed to settle in a closed flask over night, the liquid was decanted, and the precipitate was washed by decantation many times with boiling water until neutral. Cold dilute hydrochloric acid was now added, and the precipitate was digested for several days. Since the color of the supernatant liquid showed that a slight amount of even the comparatively insoluble nickel sulphide had gone into solution, the assumption was not unreasonable that nearly, if not quite, all of the more soluble sulphides must have been removed. The black precipitate was now repeatedly washed with hot water until the washings were quite



neutral; it was next dissolved in strong hot hydrochloric acid, and after the separated sulphur had been removed, the solution was evaporated to dryness, and the residue was taken up with water. The material was now considered fairly free from its usual impurities, with the single exception of cobalt.

In commenting on the work of an early experimenter upon the atomic weights of nickel and cobalt, Clarke has objected that "his results are entitled to no especial weight at present, since it cannot be certain from any evidence recorded that the oxide of either metal was absolutely free from traces of the other."\* Since the two metals have atomic weights only differing at the outside by half a unit, "traces" of one in any preparation of the other metal could not alone furnish a reason for invalidating the results. Nevertheless, for our purpose it seemed desirable to prepare nickel as nearly free from cobalt as possible. In order to attain this end with any degree of certainty, it is obvious that a qualitative test must be found that should show with sufficient accuracy the presence or absence of cobalt. Winkler† has recommended a test for which he claimed greater accuracy than the better known method with potassium nitrite. The moderately dilute solution of nickel is treated with ammonia until a clear blue color is obtained and then one or two drops of potassic permanganate are added. If no cobalt is present, the blue solution of nickel takes on a purple tinge; whereas cobalt, if present, reduces the permanganate. Winkler does not state the dilution of the permanganate solution, or how much should be added, although manifestly the degree of refinement of the test depends on these points. The permanganate solution which worked well with us contained one gram of the salt in a litre. To the dilute solution of nickel to be tested, contained in a color-comparison apparatus, enough ammonia is added to render the solution a light sky-blue, and then one tenth of a cubic centimeter of the permanganate is dropped in. Under these conditions, the mixture appears decidedly purplish in hue, if cobalt is absent. Of course the test is of value only in the absence of any foreign substances having either a reducing or an oxidizing action on permanganate. We have found it possible by this method to detect one part of cobalt in twenty-five hundred parts of nickel, an amount of impurity which could cause a final error in the atomic weight of only one part in five hundred thousand.

Anthon's process‡ for eliminating cobalt was adopted for the purification.

\* Recalculation, 1897, p. 291.

† See Dammer, *Anorg. Chem.*, III. 490.

‡ *Zeitschr. Anal. Chem.*, VI. 20.

tion of our sample. The nickel was twice fractionally precipitated as hydroxide by means of pure sodic hydroxide, the mixture being thoroughly boiled. As far as our test could show it, all the cobalt remained in the filtrate, the last precipitate being contaminated only with a small amount of alkali.

When ammonia is added to a solution of nickelous bromide, a beautiful violet crystalline compound is formed, having the formula  $\text{NiBr}_2 \cdot 6\text{NH}_3$ , according to our analyses as well as those of Rammelsberg.\* Since this compound is characteristic of nickel, and similar compounds are not formed by cobalt or most other metals under similar circumstances, and since it is soluble in strong hot ammonia water, but almost insoluble in cold ammonia, it affords a very convenient and effectual means of purifying nickel preparations. Our purified oxide was hence dissolved in pure hydrobromic acid, ammonia was added in excess, and the mixture, contained in a platinum flask, was cooled to zero. The beautiful purple precipitate was collected upon pure filter paper and was washed with strong ammonia. All the material used in the analyses was passed through this treatment at least once, although the various samples were subsequently subjected to different methods of further treatment which will be described in each case.

The violet compound made from our first sample of purified nickel was treated with an excess of water and boiled in a platinum dish, a proceeding which completely precipitated the nickel as hydroxide. The greenish mass was thoroughly washed, and was then dissolved in hydrobromic acid. The nickelous bromide thus obtained was dried in a vacuum desiccator over dry soda; but even after this treatment it was found to have retained considerable quantities of water, an impurity which greatly increased the difficulties of sublimation. Hence this sample, numbered I., was used only for two preliminary analyses.

Another portion of this same sample of the violet compound was recrystallized several times in succession by cooling its hot ammoniacal solution. The resulting magnificent crystals were dissolved in water and the solution was boiled to drive off the ammonia. The precipitation being thus accomplished the basic hydroxide was collected, carefully washed, dried, and ignited over an alcohol flame in a porcelain vessel. The resulting nickel oxide was reduced to the metal by igniting this material, held by a porcelain boat in a combustion tube through which a current of pure dry ammonia gas was passing. The spongy nickel thus

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\* Pogg. Anal., LV. 243.

produced was changed to bromide and sublimed in the manner already described. The pure substance thus prepared is designated below as number II.; it also served for two of the earlier analyses.

The further purification of the sample of nickel obtained by the process of Mond, Langer, and Quincke, at first proceeded exactly in the steps just described; except that no attempt was made to remove cobalt, since none was present. After it had reached the stage of treatment represented by the last hydroxide obtained above, the material was converted into the sulphate and subjected to electrolysis out of an ammoniacal solution in a platinum dish. The object of this procedure was of course to free the nickel still more effectually from the alkalies, silica, and many other impurities which are not precipitated on the cathode. The bright heavy deposit of pure nickel was dissolved, with a great deal of difficulty, in redistilled strong nitric acid; and the excess of acid was driven off by evaporation. Ammonia and a large excess of pure water were now added, and the solution was boiled until the basic hydroxide was completely precipitated. This was subsequently changed to metallic nickel and then into the sublimed bromide, in the manner already described, and the resulting material, labelled III., served for a large number of analyses. A small portion of it, that used for Experiment 8, was resublimed.

The sample of material used for the final analyses was even more carefully purified than this, however. A quantity of the pure nickelous oxide of about the grade of purity of sample III., coming originally from the Mond nickel, was dissolved in sulphuric acid, and the solution was made alkaline by passing in pure ammonia gas, in a platinum dish. When most of the nickel had been deposited electrolytically from this solution, the portion remaining in the electrolyte was thrown away. The bright coating of nickel was washed, pure dilute sulphuric acid was put into the dish, and with reversed poles a strong current was sent through the solution until nearly but not quite all of the nickel was dissolved. The solution was then decanted into another dish, ammonia was passed in until the precipitate formed had redissolved, the poles were again reversed, and then nickel was once more almost all deposited. This cycle of operations, which gives an excellent method of fractionation, was repeated three times. The final deposit of nickel was dissolved by filling the platinum dish with pure dilute nitric acid and reversing the poles. Only one who has tried dissolving a deposit of nickel on a platinum dish, even in strong nitric acid, can appreciate the ease, cleanliness, and convenience of this method of procedure. The solution of nickel

nitrate thus prepared was concentrated by evaporation, and ammonia was passed in until a mass of crystals of the blue ammonio-nickel nitrate was formed. After the mother liquor had been poured off, the crystals were washed with pure ammonia water, and were finally boiled in an excess of pure water in the same platinum dish. The resulting basic hydroxide was then changed to spongy nickel and nickelous bromide in the usual fashion, bearing the title No. IV.

Thus, while all of our samples of nickelous bromide analyzed had been sublimed, the several samples had received previous to sublimation very varying treatment. The fourth had been put through a process of purification far more searching than the first, which had merely been freed from the ordinary known impurities. Hence the essential identity of the results obtained from these several samples is very striking.

*Purification of other Materials.*—Silver was purified exactly in the manner described by Richards and Parker,\* in a recent paper upon the atomic weight of magnesium. The electrolytic crystals were finally fused upon a boat of pure lime in a vacuum. For further details the above mentioned paper should be consulted.

With the co-operation of Mr. Baxter, bromine was purified in a preliminary fashion by solution in strong aqueous calcic bromide, and a subsequent separation. Carefully washed red phosphorus was used to convert the bromine thus obtained, after it had been several times redistilled, into hydrobromic acid; and the hydrobromic acid was freed from iodine and organic matter by several fractional distillations with bromine water. From this pure hydrobromic acid, bromine was obtained by means of manganese dioxide free from chlorine. 2.10289 grams (in vacuum) of silver yielded 3.66066 grams (in vacuum) of argentic bromide on combination with this bromine, — a ratio of 57.445 : 100.00. Mr. Baxter found 57.444 in a similar experiment, while Stas's value was 57.445; hence the purity of our bromine and silver was proved.

Sodic hydroxide was freed from most metallic impurities (iron, etc.) by electrolysis. Ammonia was redistilled in platinum vessels, as were also nitric, and hydrochloric acids. Sulphuric acid was distilled in glass, alkalis being a less dangerous impurity than platinum in the instances where it was used. Water was purified by distillation first from alkaline permanganate solution, and then with a trace of acid potassic sulphate.

In processes where the presence of bromine rendered the use of platinum impossible, Jena glass, or at high temperatures Berlin porcelain,

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\* These Proceedings, XXXII. 62.

was used. For some of the platinum and other apparatus we are indebted to the Cyrus M. Warren Fund for chemical research in Harvard University.

#### THE METHOD OF ANALYSIS.

Turning now to the method of analyzing the carefully prepared nickelous bromide, it is obvious that the first point to be considered is the accurate determination of the weight of the salt to be analyzed. This process was effected by means of apparatus similar to that devised for the drying and weighing of magnesian chloride, and described in a recent paper by Mr. H. G. Parker and one of us,\* upon the atomic weight of magnesium. In this apparatus, constructed wholly of glass by Mr. Baxter, the bromide under consideration, contained in a platinum boat, was ignited at about  $400^{\circ}$  in a stream of mixed nitrogen and hydrobromic acid until

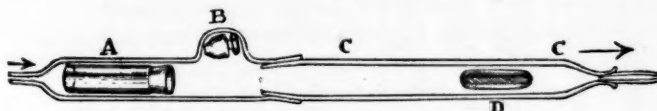


FIG. 2. BOTTLING APPARATUS, HORIZONTAL SECTION.

A = weighing bottle. B = stopper of bottle. CC = hard glass tube.  
D = Platinum boat containing nickelous bromide.

constant in weight. It was then allowed to cool in a stream of pure dry nitrogen; and when cool it was pushed in pure dry air into its weighing bottle, which was immediately closed by a mechanical device. In this fashion it is possible to dry and weigh accurately the most hygroscopic of substances; and repeated ignitions of the same specimen have shown that perfect constancy in weight may thus be obtained. It is hard to believe that any water is retained by nickelous bromide at  $400^{\circ}$ ; and certainly none could be absorbed during the cooling, for the whole apparatus was shut off from the outside air, and all the gases admitted were first passed through phosphoric oxide.†

The bromide in question was then weighed by substitution, using as the tare to be substituted a weighing bottle precisely like the one containing the platinum boat and substance. In this way alone can the weight of a large bottle be determined within the fraction of a tenth of a

\* These Proceedings, XXXII. 58.

† A detailed description of this apparatus will be given in the following paper, upon Cobalt.

milligram. The balance has already been described in detail;\* the weights were of course compared and standardized with great care, and were used for no other work during the progress of this.

Having been weighed with accuracy, the nickelous bromide was dissolved in pure warm water in a flask, and from this was transferred to the large beaker flask in which the precipitation was to take place. The platinum boat in which the salt had been treated remained invariable in weight, showing that it had not been attacked by hydrobromic acid at a high temperature.

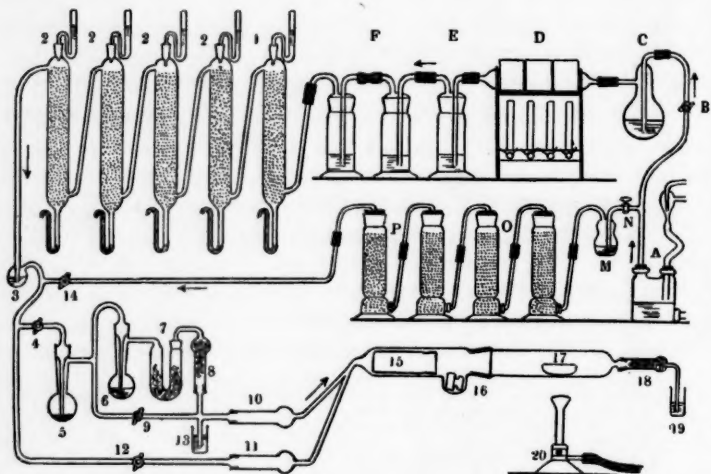


FIG. 3. APPARATUS FOR IGNITING NICKELOUS BROMIDE IN ANY DESIRED MIXTURE OF GASES.

The use of rubber was confined to the first part of this train, where it could do no harm (A B C D E F and A M N O P).

As has been said already, the salt used in the preliminary series was contaminated with a small amount of nickelous oxide, which was filtered off and weighed. The amount of this impurity is given simply to show that the slight irregularity of the results was not dependent upon the adulteration; the weights of nickelous bromide given are those left after the subtraction of the weight of the oxide. All the bromine contained in the solution was precipitated in these seven analyses by means of an

\* These Proceedings, XXVI. 242.



excess of argentic nitrate, and the argentic bromide was collected and weighed with the usual precautions.

When the manipulation had thus been mastered, the art of preparing absolutely pure nickelous bromide had been perfected (see Analysis 7), and the atomic weight of nickel had been approximately determined, the method of procedure in subsequent analyses was changed. The now perfectly clear solution was treated with just enough argentic nitrate, prepared from the purest weighed silver, to complete the precipitation. The mean between the two possible end points was determined by titrating backwards and forwards with hundredth normal argentic nitrate and hydrobromic acid solutions;\* and thus was determined the ratio of silver to nickelous bromide entitled Series III. After this end point had been determined, a slight excess of argentic nitrate was added to the solution, and the whole was violently shaken. The precipitate was collected upon a Gooch crucible, washed with water containing a trace of argentic nitrate, later with pure water, and finally dried and weighed. The traces of asbestos carried away by the wash water were of course determined, and all the usual precautions were taken to insure great accuracy. Thus was obtained the series of results given in Series II.

#### THE ATOMIC WEIGHT OF NICKEL.

O = 16.000; Ag = 107.93.

FIST SERIES (PRELIMINARY). RATIO = 2 AgBr : NiBr<sub>2</sub>.

Number of Experiment.	Sample of NiBr <sub>2</sub> .	Weight of Nickelous Bromide in Vacuum.	Weight of Insoluble Residue.	Weight of Argentic Bromide in Vacuum.	Atomic Weight of Nickel.
1	I.	grams. 2.26113	milligrams. 3.22	grams. 3.88769	58.646
2	I.	2.80608	7.08	4.82431	58.708
3	II.	1.41317	3.05	2.42880	58.716
4	II.	1.71759	0.88	2.95307	58.650
5	III.	2.48565	5.24	4.27357	58.651
6	III.	4.32997	15.83	7.44280	58.700
7	III.	2.18072	0.00	3.74856	58.693
Average . . . . .					58.680

\* For the details see These Proceedings, XXX. 384.



SECOND SERIES: RATIO = 2 AgBr : NiBr<sub>2</sub>.

Number of Experiment.	Sample of NiBr <sub>2</sub> .	Weight of Nickelous Bromide in Vacuum.	Weight of Argentic Bromide in Vacuum.	Atomic Weight of Nickel.
8	III.	grams. 3.28039	grams. 5.63892	58.691
9	III.	2.70044	4.64208	58.686
10	III.	3.38230	5.81391	58.698
11	III.	1.33459	2.29435	58.670
12	IV.	1.25054	2.14963	58.693
13	IV.	1.32278	2.27384	58.690
14	IV.	2.24452	3.85805	58.705
Average . . . . .				58.690

THIRD SERIES: RATIO = 2 Ag : NiBr<sub>2</sub>.

Number of Experiment.	Sample of NiBr <sub>2</sub> .	Weight of Nickelous Bromide in Vacuum.	Weight of Silver in Vacuum.	Atomic Weight of Nickel.
8	III.	grams. 3.28039	grams. 3.23910	58.701 *
9	III.	2.70044	2.66636	58.709
10	III.	3.38230	3.33990	58.689
11	III.	1.33459	1.31787	58.689
12	IV.	1.25054	1.23482	58.698
13	IV.	1.32278	1.30629	58.675
14	IV.	2.24452	2.21652	58.676
Average . . . . .				58.691

In the table above, the first column records the number of the experiment, the second records the number of the sample of nickelous bromide used, while the third records the weight of this salt taken. The extreme right hand column contains the atomic weight of nickel computed from the values contained in the one just to the left of it, and those contained in the third.

A very interesting evidence of the accuracy of these results is the relationship between the amount of silver taken and the amount of argentic bromide obtained. From the second and third series, we find that 15.51556 grams of nickelous bromide yielded 26.67078 grams of argentic bromide, requiring 15.32086 grams of silver. This leads to the inference that argentic bromide contains 57.444 per cent of silver, a quantity which agrees essentially with the value 57.445 per cent found by Stas. Since the bromine used had been already found to be free from other halogens, and the silver was known to be perfectly pure, we have in these results conclusive proof that no nickel salt was occluded by the argentic bromide, as well as a satisfactory "check" upon the accuracy of the work.

When we examine the results with respect to the various samples of the salt analyzed, we find a very interesting and satisfying uniformity. The four samples of nickelous bromide gave the following results for the atomic weight of nickel:—

Sample I.	58.677
Sample II.	58.683
Sample III.	58.688
Sample IV.	58.689

The slight rise in the value with increasing purity is not large enough to have any weight, for there are analyses in the lowest series giving higher individual results than any in the highest series. Hence we are forced to the conclusion, that the least carefully purified specimens of nickelous bromide must have been essentially identical with the most carefully purified. The chances are evidently exceedingly small that the impurities would so combine as exactly to counterbalance one another.

The further discussion of this important question will be reserved until more experimental work has been done. For the present, it is our opinion, at this first halting place in a long investigation, that the atomic weight of nickel cannot be far from 58.69 if  $O = 16.00$ , or 58.25 if  $O = 15.88$ .